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IS 3999 (1966): Casein Based Aqueous Pigments and Finishes
[CHD 17: Leather, Tanning Materials and Allied Products]



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Indian Standard

SPECIFICATION FOR
CASEIN BASED AQUEOUS PIGMENTS
AND FINISHES

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

SPECIFICATION FOR CASEIN BASED AQUEOUS PIGMENTS AND FINISHES

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Indian Standard

SPECIFICATION FOR CASEIN BASED AQUEOUS PIGMENTS AND FINISHES

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 1 October 1966, after the draft finalized by the Leather Sectional Committee had been approved by the Chemical Division Council.

0.2 Casein based aqueous pigments are used widely in the country along with casein based binders and synthetic polymeric lattices. The water dispersible pigments, that is, the basic colouring matter in aqueous finishes, are prepared by dispersing dry pigment colours or pulps, such as synthetic, precipitated inorganic or organic pigments and earth colours in casein solutions with suitable amount of plasticizers and other ingredients, such as thickeners or agents, to impart desired gloss. To contribute some specific properties to the finish other subsidiary binders are also added. These pigments which are generally in thick viscous paste form are prepared in a variety of colours and shades. The general characteristics like viscosity; total solid; covering power; protein content; preservatives and oils, fats, and waxes used in the manufacture, differ from one manufacturer to another. Some manufacturers market these pigments in a ready-to-use form for direct application to the leather with or without extra casein based binders which are marketed separately for finishing different types of leather, others market pigments and binders separately with their own auxiliary finishing agents to be used in proportions recommended by them. The amounts of the binders and pigments to be used in the finishing of different types of leathers also vary considerably. There are still some consumers who use the commercial pigments and prepare their own binders for use admixed with synthetic or other types of binders.

0.3 In all these cases, however, the pigments and binders have to be used essentially in the finishing of different types of leathers used for goods of daily use like shoe uppers, case sides and portfolio leathers. Essentially the finishes, especially the pigment pastes and binders commonly used, shall be of a standard quality. The comparison of two brands of pigment pastes on the basis of the analytical figures will not indicate

any idea about the quality of the finish, as it is only the final formulation at the hands of the finisher that mostly decides the quality of the finish. As such, the best way to judge the performance of the finish is to finish the leather as recommended by the manufacturer and then test for the various finishing properties, according to the methods prescribed in this specification.

0.4 In view of the complexity of the types of finishes the formulation of this standard has been approached from the practical point of view and stress has been laid on physical and practical tests in preference to chemical tests which fail to give any clue as to the satisfactory nature of the products.

0.5 The basic raw materials, pigment colours (organic or inorganic) used in the manufacture of the pigment paste, would be of a quality, normally as specified under the corresponding Indian Standard specifications for oil paints (not for all items) or inks or both which may be taken for guidance.

0.5.1 In the manufacture of these finishes and binders, use of lactic casein conforming to IS: 167-1957*, shellac conforming to grade A of IS: 16-1956† and Turkey red oil conforming to IS: 1044-1957‡ is recommended.

0.6 In the preparation of this standard considerable assistance has been derived from the experience of Central Leather Research Institute, Madras and from B.S. 3662: 1962 'Methods for the determination of the colour fastness of the leathers', issued by the British Standards Institution.

0.7 This standard contains clauses 3.2, 3.6, 3.7, 6.2 and 6.6 which call for agreement between purchaser and supplier and which require the purchaser to supply certain technical information at the time of placing orders.

0.8 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS: 2-1960§. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Specification for casein (*revised*).

†Specification for shellac (*revised*).

‡Specification for Turkey red oil.

§Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the requirements, methods of sampling and test for casein based aqueous pigment finishes and casein based binders with or without dispersed pigments for leather.

1.2 This standard does not cover the so called dispersed pigments or concentrated pigment colours which do not contain any casein binder and which are generally meant for use with synthetic resin emulsions or other commercially known synthetic resin binders used in the leather industry.

2. TERMINOLOGY

2.1 For the purposes of this standard, the definitions given in IS: 1640-1960*, shall apply.

3. REQUIREMENTS

3.1 Physical Appearance and Nature of the Product — The pigment paste shall be homogeneous, uniform, of viscous consistency, smooth to touch when rubbed between two fingers or should have no undispersed or agglomerated particles, caking of the pigment in the container and shall be easily dispersible when mixed with water. On storage there shall be no sedimentation that shall not be readily dispersed by gentle stirring or shaking.

3.2 Uniformity — All lots in a single consignment shall agree in moisture content, viscosity and colour with each other and with the approved sample previously agreed to between the purchaser and the supplier.

3.3 The particle size of the pigment dispersion shall be of the order of $2.5\text{ }\mu\text{m}$ to $5\text{ }\mu\text{m}$ with exception of a few stray agglomerates not exceeding $25\text{ }\mu\text{m}$, when determined using a fineness meter or when tested under a microscope.

3.4 The casein binder shall be homogeneous, uniform in consistency without showing any signs of sedimentation, and shall be opalescent. The film dried from the binder shall be clear.

3.4.1 The casein binder shall have a pH range 6 to 8.5 on dilution with five times its water, determined electrometrically.

3.5 During the manufacture of the binder and the pigment paste, necessary quantities of preservative for the casein shall be incorporated so as to ensure a shelf life of at least one year, if stored in closed containers in which the material had been originally received by the purchaser and stored in cool and dry place.

*Glossary of terms relating to hides, skins and leather.

3.6 The shade and the covering power (*see* 8 of IS : 101-1964*) shall be as agreed to between the purchaser and the supplier.

3.7 The pigment finish shall be applied in accordance with the manufacturer's instructions to test pieces of chrome tanned and dyed crust leather (*see* IS : 578-1964†) and vegetable tanned skin ready for finish (*see* IS : 1639-1960‡) and shall be tested for the various finishing properties as given in Table 1.

TABLE 1 REQUIREMENTS FOR FINISHING PROPERTIES OF AQUEOUS CASEINOUS PIGMENTS AND FINISHES

SL No.	CHARACTERISTIC	REQUIREMENTS	METHOD OF TEST REF TO CL
(1)	(2)	(3)	(4)
i)	Wet and dry rub resistance	To pass test	26 of IS : 582-1964*
ii)	Nature of the finished film	The film shall not crack, peel or crumble	—
iii)	Gloss of the finished leather	Shall have the same degree of gloss previously agreed to between the purchaser and the supplier	6.5
iv)	Resistance to lasting	To pass test	6.7
v)	Fastness to day light	To pass test	6.8
vi)	Fastness to organic solvents	To pass test	6.9
vii)	Fastness to water spotting	To pass test	6.10
viii)	Resistance to heat	On hot plating or ironing it shall comply with requirements (i), (ii), (vi) and (vii) when tested after hot plating or ironing	
ix)	Ageing	On ageing the finished test piece as in 3.10 at $70^{\circ} \pm 1^{\circ}\text{C}$ for one hour and subsequent cooling to room temperature, the piece shall comply with the requirements (i), (ii), (v), (vi) and (vii)	

*Methods of sampling and test for vegetable and chrome tanned leathers.

*Methods of test for ready mixed paints and enamels (*second revision*).

†Specification for full-chrome upper leather (*revised*).

‡Specification for East India tanned kips and skins.

4. PACKING AND MARKING

4.1 Packing — The material after manufacture shall be packed in tight containers which shall not develop any rust or deterioration of the product due to the presence of water in the medium or any of the ingredients used in the formulation.

4.2 Marking — The containers shall be marked with the name of the manufacturer, trade-mark, if any; batch number, date of manufacture, shade and the quantity.

4.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act, and the Rules and Regulations made thereunder. Presence of this mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard, under a well-defined system of inspection, testing and quality control during production. This system, which is devised and supervised by ISI and operated by the producer, has the further safeguard that the products as actually marketed are continuously checked by ISI for conformity to the standard. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING AND CRITERIA FOR CONFORMITY

5.1 Scale of Sampling — For the purpose of ascertaining the conformity of the material in any consignment to this specification, the scale of sampling and criteria for conformity shall be as prescribed in 3 of IS : 101-1964*

6. TEST METHOD

6.1 The samples of the pigment or binder or both shall be tested for finishing properties (*see* Table 1) by application on leather pieces, chrome tanned and dyed crust leather (*see* IS : 578-1964†) and vegetable tanned skin ready for finish (*see* IS : 1639-1960‡), 30 cm × 30 cm pieces. The pigments shall be tested using a casein binder to be supplied by the manufacturer or prepared according to the recipe, furnished by the manufacturer.

6.2 The leather pieces shall be finished as per detailed method to be furnished by the manufacturer using the auxiliaries as suggested by him. All the ingredients shall be of the quality as agreed to between the purchaser and the supplier, wherever such requirements are not prescribed for the ingredients in this standard.

*Methods of test for ready mixed paints and enamels (*second revision*).

†Specification for full-chrome upper leather (*revised*).

‡Specification for East India tanned kips and skins.

6.3 At least three test pieces of each type of leather shall be tested.

6.4 The material shall be tested for wet and dry rub resistance in accordance with the methods of test prescribed in IS : 582-1954*.

6.5 Tests for Uniformity — The material shall be tested in accordance with the method prescribed in 7.4 of IS : 101-1964† for viscosity and in 26 of IS : 101-1964† for volatile matter.

6.6 Gloss of the Finished Leather — The gloss of the finished leather shall be tested by a sheen gloss meter (see IS : 101-1964†) or any other device as agreed to between the purchaser and the supplier.

6.7 Resistance to Lasting — A key drawn against the leather from the flesh side by holding the leather firm against key between the thumb and the first finger shall show no signs of crackiness or peeling of pigment film. The finished leather shall show no sign of crackiness or peeling of pigment film at the double fold.

6.8 Fastness to Daylight — The test pieces finished with the pigment finish and dried, shall be tested for fastness to daylight in accordance with the method prescribed in IS : 686-1957‡ except that the material shall be finished leather. The leather under test and the standards, which are exposed simultaneously shall be partially covered with an opaque card and exposed to daylight. They shall be protected from the weather by exposing them under window glass facing due south and sloping at an angle of 45°, care being taken to ensure that obstructions do not cast shadows on the pattern. Adequate ventilation shall be provided.

6.8.1 Assessment — Fastness to rating shall be 3 to 4 in the case of vegetable tanned leather and 4 or better for chrome tanned leather.

6.9 Fastness to Organic Solvents

6.9.1 Outline of the Method — A sample of the leather under test is wetted from the back with a felt pad soaked in the chosen solvent or mixture of solvent. The grain-surface is then tested for fastness to dry and wet rubbing.

6.9.2 Reagents

6.9.2.1 Solvent mixture — The three types of solvent mixtures

*Methods of sampling and test for vegetable and chrome tanned leathers.

†Methods of test for ready mixed paints and enamels (*second revision*).

‡Method for determination of colour fastness of textile materials to daylight.

depending on their nature of drying, shall be as follows:

Percent by Volume

i) Mixture A (slow drying):	
Ethyl methyl ketone	35
Iso-propyl alcohol (<i>see</i> IS : 2631-1964*)	30
Diacetone alcohol (<i>see</i> IS : 2252-1962†)	20
ii) Mixture B (medium drying):	
Ethyl methyl ketone	40
Ethyl acetate (<i>see</i> IS : 229-1957‡)	30
Ethanol (<i>see</i> IS : 324-1959§)	30
iii) Mixture C (fast drying):	
Acetone (<i>see</i> IS : 170-1953¶)	70
Methanol (<i>see</i> IS : 517-1954)	30

NOTE — Any one, two or all the three mixtures shall be agreed upon between the purchaser and the supplier prior to testing for this characteristic.

6.9.2.2 Felt pad — 25 mm square piece, 6 mm thick, medium soft white felt.

6.9.3 Procedure — Wet the felt piece evenly with 2.5 ± 0.1 ml of the solvent mixture delivered by pipette. Wet the back portion of the test piece of leather uniformly with the wetted felt. Hang the test pieces to dry in air at room temperature. Test for dry and wet fastness of the finish of the leather at the tested areas as described in 26 of IS : 582-1954**.

6.9.4 Results — The finish shall not crack, or peel or crumble and shall be fast to wet and dry rubbing (*see* IS : 582-1954**).

6.10 Fastness to Water Spotting

6.10.1 Outline of the Method — One drop of distilled water is placed on the finished side of the leather; after 30 minutes any surplus water is removed with filter paper and physical effects, if any, are observed.

*Specification for iso-propyl alcohol.

†Specification for diacetone alcohol.

‡Specification for ethyl acetate.

§Specification for ethanol.

¶Specification for acetone.

||Specification for methanol.

**Methods of sampling and test for vegetable and chrome tanned leathers.

6.10.2 Apparatus — Pipette or dropper to deliver approximately 0.15 ml of water.

6.10.3 Procedure — Place drops of distilled water on the specimen about 5 cm apart. After 30 minutes remove the residual water (if any) by gently blotting with filter paper and observe the physical effects.

6.10.4 Reporting — There shall be no swelling, loss of lustre or any visible change when compared with the original spot.

NOTE — It may be necessary to tilt the leather in all directions during observation and the magnitude of the effect may be described as slight, moderate or severe in comparison to the original sample. Polishing lightly with a nonstaining wax polish is permissible after water treatment prior to assessment.